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A series of monoazido alkanes and diazido alkanes have been synthesized. Specifically, 1-azidobutane (100.0 g), 1-azidopentane (100.0 g), 1-azidohexane (100.0 g), 1-azidooctane (100.0 g), and 1-azidoheptane (100.0 g), 1,3-diazidopropane (200.0 g), 1,4-diazidobutane (200.0 g), 1,5-diazidopentane (200.0 g), 1,6-diazidohexane (200.0 g), 1,7-diazidoheptane (200.0 g), 1,8-diazidooctane (200.0 g) were synthesized and characterized completely both analytically and spectroscopically. Among cyclic examples 1,2-diazidocyclopentane, 1,2-diazidocyclohexane and 1,4-diazocyclohexane were synthesized. Norbornene was converted into a regioisomeric mixture of 2,3-diazido and *syn* and *anti*-2,7-diazidonorbornanes. The photodimer of norbornadiene was synthesized and converted to the tetraazido analog. These compounds were transmitted to C.K. Law, Princeton, for droplet combustion experiments. Dihydrobenzvalene was synthesized and preliminary developmental work has been initiated. A sample has been transmitted to C.K. Law for combustion experiments.

20. DISTRIBUTION/AVAILABILITY OF ABSTRACT

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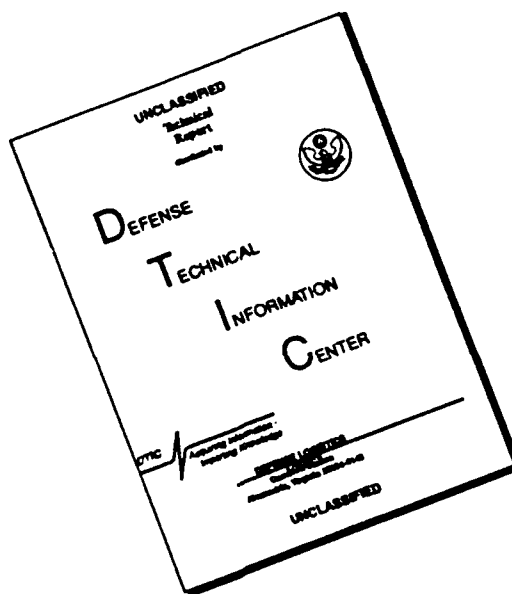
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**ALKYL AZIDES, DIAZIDES, HALOAZIDES AND BRIDGED  
POLYCYCLIC DIAZIDES**

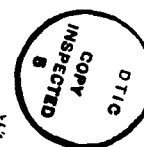
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## **Abstract**

A series of monoazido alkanes and diazido alkanes have been synthesized. Specifically, 1-azidobutane (100.0 g), 1-azidopentane (100.0 g), 1-azidohexane (100.0 g), 1-azidooctane (100.0 g), and 1-azidoheptane (100.0g), 1,3-diazidopropane (200.0 g), 1,4-diazidobutane (200.0 g), 1,5-diazidopentane (200.0 g), 1,6-diazidohexane (200.0 g), 1,7-diazidoheptane (200.0 g), 1,8-diazidooctane (200.0 g) were synthesized and characterized completely both analytically and spectroscopically. Among cyclic examples 1,2-diazidocyclopentane, 1,2-diazidocyclohexane and 1,4-diazocyclohexane were synthesized. Norbornene was converted into a regioisomeric mixture of 2,3-diazido and syn and anti-2,7-diazidonorbornanes. The photodimer of norbornadiene was synthesized and converted to the tetraazido analog. These compounds were transmitted to C.K. Law, Princeton, for droplet combustion experiments.

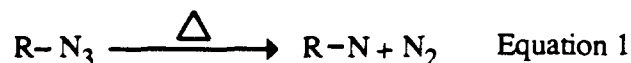
Dihydrobenzvalene was synthesized and preliminary developmental work has been initiated. A sample has been transmitted to C.K. Law for combustion experiments.

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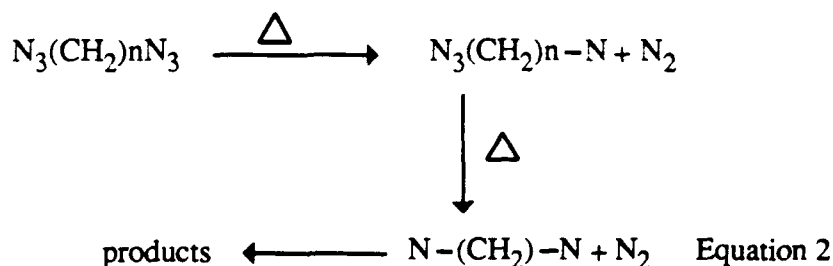
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## 1. Introduction

In continuation of our original concept, namely, the thermal decomposition of alkyl azides in the liquid phase of droplets exothermically releases nitrogen, we extended the scope of work to include long chain diazides and strained polycyclic polyazides. The decomposition of a monoazide is represented by equation 1:



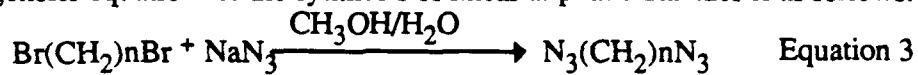
In the case of a diazide the release of nitrogen would be stepwise:



In the case of a bridged polycyclic system the strain energy of the carbocyclic framework would contribute additionally to the exothermic fragmentation.

## Method of Synthesis

The generic equation for the synthesis of linear aliphatic diazides is as follows:



$$n = 4, 5, 6, 7, 8, 9$$

Table 1 presents a list of the various alkyl diazides which have been synthesized.

**Table I Mono and Diazidoalkanes Synthesized**

**A. Monoazidoalkanes**

1)	1-Azidobutane	[100.0 g]
2)	1-Azidopentane	[100.0 g]
3)	1-Azidohexane	[100.0 g]
4)	1-Azidoheptane	[100.0 g]
5)	1-Azidooctane	[100.0 g]

**B. Diazidoalkanes**

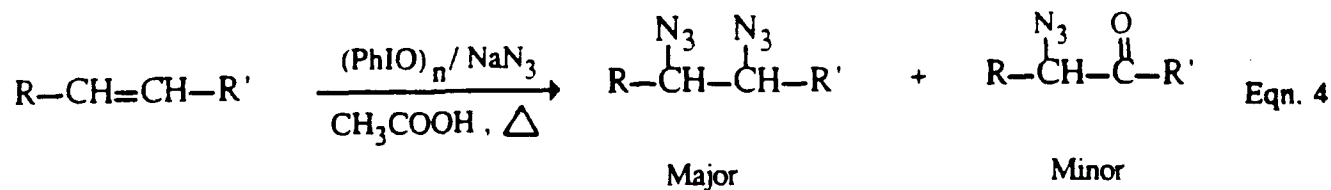
1)	1,3-Diazidopropane	[200.0 g]
2)	1,4-Diazidobutane	[200.0 g]
3)	1,5-Diazidopentane	[200.0 g]
4)	1,6-Diazidohexane	[200.0 g]
5)	1,7-Diazidoheptane	[200.0 g]
6)	1,8-Diazidooctane	[200.0 g]

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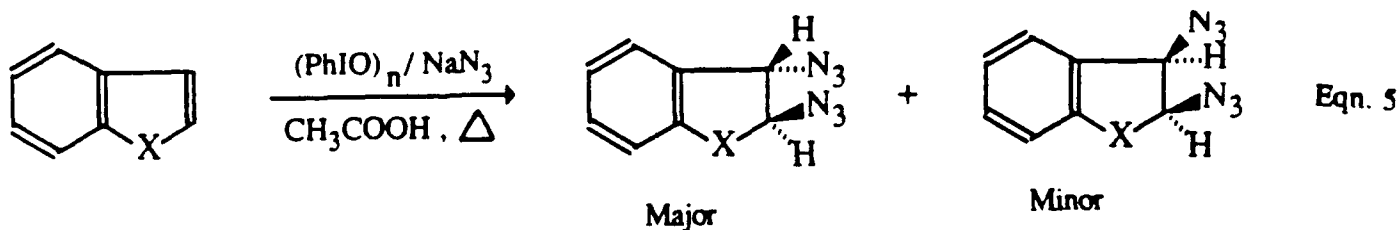
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## 2. Synthesis of Vicinal Diazides. Hypervalent Iodine Method

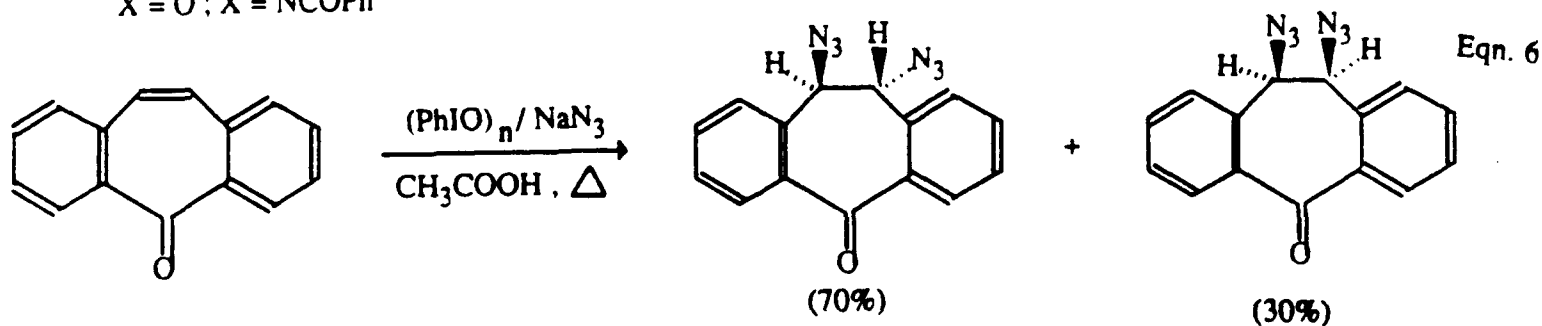
In this process (Eqn 4) a double bond is converted into a 1,2-diazide:



R = R' = H or alkyl or aryl



X = O ; X = NCOPh

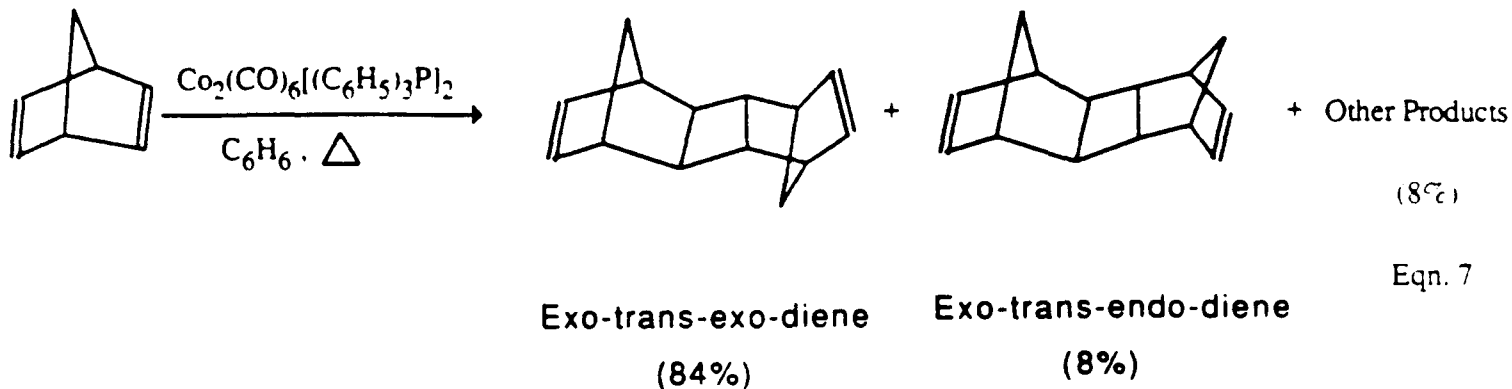


Using this method we have synthesized some cycloalkyl diazides which will be evaluated by CK Law in combustion experiments. Specifically cyclopentene and cyclohexene have been converted to 1,2-diazidocyclopentane and 1,2-diazidocyclohexane, respectively.

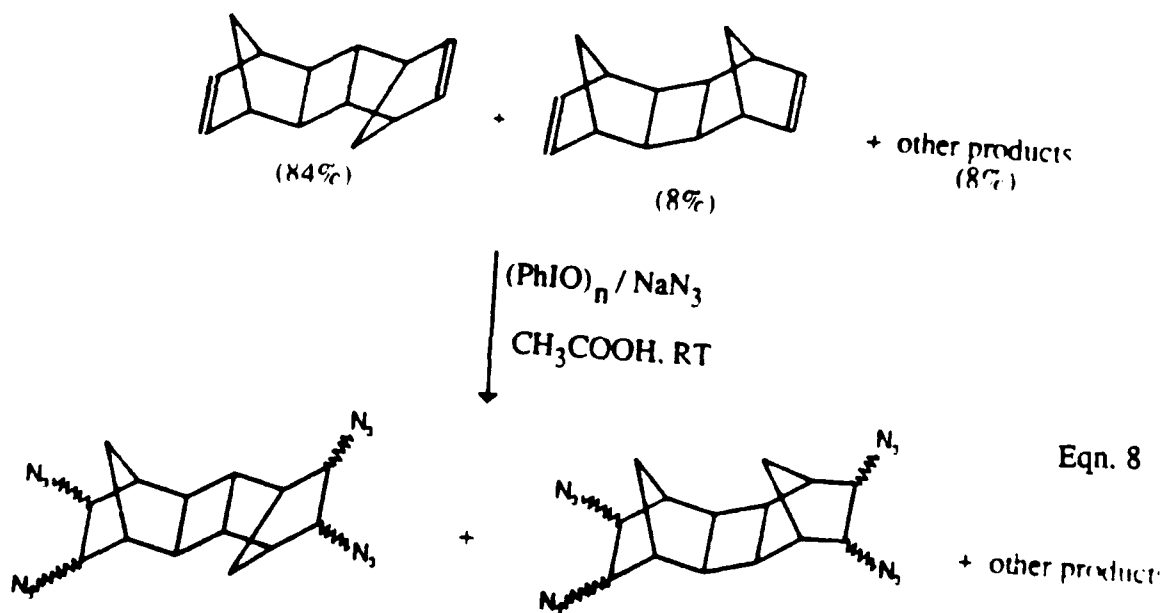


### 3. Synthesis of Norbornadiene Dimers

Using the method shown in Eqn. 7 norbornadiene was converted into exo-trans-exo and exo-trans-endo dimer.



The dimer mixture was purified by column chromatography and converted directly to a mixture of the corresponding tetraazido derivatives.



The mixture of isomeric tetraazides was transmitted to CK Law for combustion experiments.

4. **References:**

1. R.M. Moriarty and J. Khosrowshahi, *Tetrahedron Lett.*, **27**, 2809, 1986.
2. D.R. Arnold, D.J. Trecker, and E.B. Whipple, *J.Am.Chem. Soc.*, **87**, 2596, 1965.

## 5 **EXPERIMENTAL**

### **Synthesis of Monoazidoalkanes**

#### General Procedure:

1-Bromooctane [96.5 g, 0.5 mol] was dissolved in methanol (500 ml). To this solution sodium azide [65.0g, 1.0 mol] in water (300 ml) was added in one portion and the contents were refluxed for 2 days. The reaction mixture was cooled to room temperature and concentrated in vacuo to ~ 400 ml. Water (500 ml) was added to the concentrate and extracted with ether (3 x 500 ml), dried over anhydrous magnesium sulfate, filtered and concentrated in vacuo to yield the product (70. g, 90%).

### **Synthesis of Diazidoalkanes**

#### General Procedure:

1,4-Dibromobutane [64.5 g, 0.3mol] was dissolved in methanol (500 ml). To this solution sodium azide [97.5 g, 1.5 mol] in water (300 ml) was added in one portion and the contents were refluxed for 2 days. The reaction mixture was cooled to room temperature and concentrated in vacuo to ~400 ml. Water (500 ml) was added to the concentrate and extracted with ether (3 x 500 ml), dried over anhydrous magnesium sulfate, filtered and concentrated in vacuo to yield the product [40.0 g, 95%].

### **Synthesis of Norbornadiene Dimers**

Norbornadiene [135.0 g, 1.46 mole], hexacarbonyl bis(triphenyl phosphine) dicobalt [3.0g 0.0037 mole] and benzene (120 ml) were stirred together under reflux for 48 hours. The reaction mixture was cooled to room temperature and then washed with dil HCl [2N, 500ml] and separated, and the aqueous portion was extracted with ether (2 x 500 ml). The combined organic portions were washed with saturated sodium carbonate (2 x 500 ml), dried over anhydrous  $\text{MgSO}_4$ , filtered and concentrated in vacuo to yield the product (98.0g, 73%).

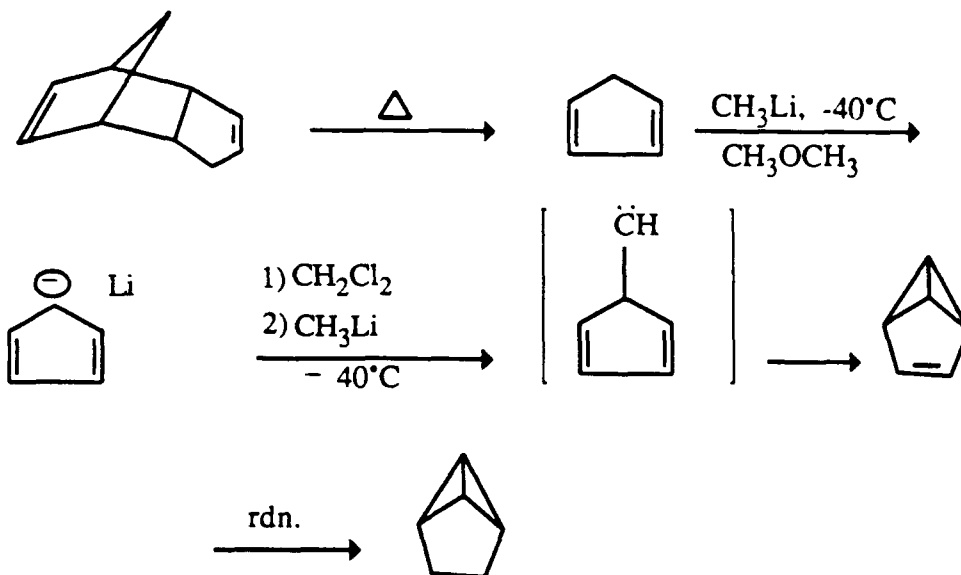
### **Synthesis of Polyazidonorbornane Dimers**

Iodosobenzene [26.4 g, 0.12 mol] was added to acetic acid (500 ml) followed by the addition of norbornadiene dimers [9.2 g, 0.05 mol] at 0° C. The reaction mixture was stirred at 0° C for 15 min., then sodium azide [26.0 g, 0.4 mol] was added to the reaction

mixture and the contents were heated at 40° C for 2 hours. The reaction mixture was cooled to room temperature and then poured into water (1000 ml) and extracted with chloroform (3 x 500 ml). The organic phase was then washed with saturated sodium bicarbonate solution (3 x 500 ml), dried over anhydrous  $\text{MgSO}_4$ , filtered, concentrated in vacuo to give the crude product (30 g). The crude product was then passed through a silica gel column using 20% ethyl acetate in hexane as an eluant. Yield 8.0 g.

## 6. Dihydrobenzvalene

Work was initiated during the current project on the synthesis of dihydrobenzvalene.



During the contract period this synthesis was standardized, and currently we are producing 30g/wk of 80:20% dihydrobenzvalene:benzene.

## 7. Preparation of Dihydrobenzvalene

Methylolithium (457.2 ml, 640 mmol, Aldrich, 1.4 M solution in diethyl ether) was cannulated into a dropping funnel (500 ml) under an  $N_2$  atmosphere. The methylolithium was slowly introduced into a 5 L three necked flask. An aspirator outlet was adopted to remove ethyl ether from the methylolithium. After all the ethyl ether was removed under reduced pressure, dry  $N_2$  gas was introduced and the flask was cooled to  $-78^\circ C$  (dry ice-acetone bath). Methyl ether gas was introduced slowly through the adaptor while cooling until approximately 1200 ml of methyl ether was collected. The dry ice-acetone bath was replaced with a dry ice-acetonitrile bath ( $-42^\circ C$ ). Freshly distilled cyclopentadiene (40 g, 605 mmol) was slowly added from a dropping funnel into the stirred solution. After vigorous evolution of methane subsided, dry dichloromethane (104 g, 1.22 moles) was added dropwise with stirring. The slurry turned yellow. An ether solution containing methyl lithium (900 ml, 1.26 moles, Aldrich, 1.4 M solution in diethyl ether) was added very slowly with stirring. After complete addition, the solution was slowly warmed to room temperature. Most of the methyl ether was removed during this process. The ethyl ether was distilled from the reaction mixture using a water aspirator into a receiver cooled in a dry ice-acetone bath. The benzvalene was codistilled with ethyl ether and some benzene. (Note: distillation was carried out until all of the ether was removed. The ether solution of benzvalene was used as such for the reduction without further purification).

### Step II

Hydrazine hydrate (175 g, 3.5 mol), ethanol amine (175 g, 2.87 mol), and 2-methoxy ethanol (750 ml, 9.51 mol) were added to approximately 18 g of benzvalene (230.8 mmol) which was in 1500 ml of ether and methylene chloride. An aqueous solution of potassium ferricyanide (385 g, 1.17 mol, dissolved in 900 ml of water) was added dropwise with stirring (Note: a mechanical stirrer was used). During the reaction,  $N_2$  gas was eliminated. The gas was passed through a condenser cooled with dry ice-acetone ( $-78^\circ C$ ). Most of the volatiles were condensed and went back into the mixture. After complete addition of the potassium ferricyanide solution, the mixture was stirred for an additional 30 minutes. The total time of the reduction was 4-5 hours. Then the ether was distilled using a water aspirator into a receiver cooled in a dry ice-acetone bath. While distilling, slowly warm the solution with hot water until a temperature of  $35^\circ C$  is reached. Wash the ether layer with distilled water (3 x 100ml) and dried over anhydrous sodium sulfate. The dried ether was then distilled. The dihydrobenzvalene comes with ether and methylene chloride at  $35-60$ ,  $60-70$  and  $70-75^\circ C$ . All the fractions were again fractionally distilled. (Note: most of the benzene was removed from the reaction mixture by first

freezing and then by distillation of the decanted liquid).

Overall yield from cyclopentadiene: 12.9 g, 27%

B.P.: 69-70 °C;  $^1\text{H}$  NMR  $\delta$  1.25 (s, 4H), 1.67 (br. s, 2H) and 2.05 (br. s, 2H). NMR spectral data agrees with the reported data in literature.<sup>1</sup>

<sup>1</sup> M. Christl and G. Bruntrup, *Chem. Ber.*, **107**, 3908 (1974).

8. **ADENDUM**

1.  $^1\text{H}$  NMR [400 MHz] OF 1,5-DIBROMOPENTANE
2.  $^{13}\text{C}$  NMR [100 MHz] OF 1,5-DIBROMOPENTANE
3. IR SPECTRUM OF 1,5-DIBROMOPENTANE
4.  $^1\text{H}$  NMR [400 MHz] OF 1,5-DIAZIDOPENTANE
5.  $^{13}\text{C}$  NMR [100 MHz] OF 1,5-DIAZIDOPENTANE
6. IR SPRECTRUM OF 1,5-DIAZIDOPENTANE
7. HPLC OF 1,5-DIAZIDOPENTANE
8.  $^1\text{H}$  NMR [400 MHz] OF 1,6-DIBROMOHEXANE
9.  $^{13}\text{C}$  NMR [100 MHz] OF 1,6-DIBROMOHEXANE
10.  $^1\text{H}$  NMR [400 MHz] OF 1,6-DIAZIDOHEXANE
11.  $^{13}\text{C}$  NMR [100 MHz] OF 1,6-DIAZIDOHEXANE
12. MASS SPECTRUM OF 1,6-DIAZIDOHEXANE
13. IR SPECTRUM OF NORBORNADIENE DIMERS
14.  $^1\text{H}$  NMR [400 MHz] OF NORBORNADIENE DIMERS
15. IR SPECTRUM OF TETRAAZIDONORBORNANE DIMERS
16.  $^1\text{H}$  NMR [400 MHz] OF TETRAAZIDONORBORNANE DIMERS
17. MASS SPECTRUM OF TETRAAZIDONORBORNANE DIMERS
18.  $^1\text{H}$  NMR OF DIHYDROBENZVALENE
19. GAS CHROMATOGRAPH OF DIHYDROBENZVALENE



# <sup>1</sup>H NMR [400 MHz] of 1,5-Dibromopentane



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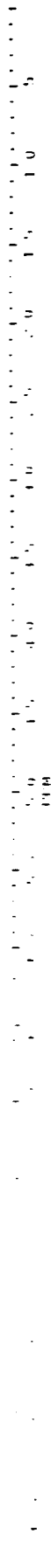
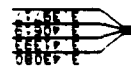
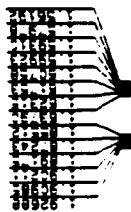
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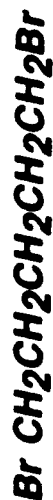
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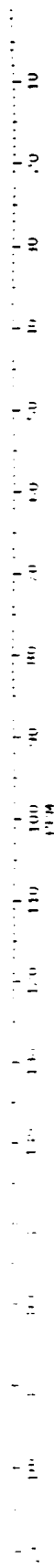
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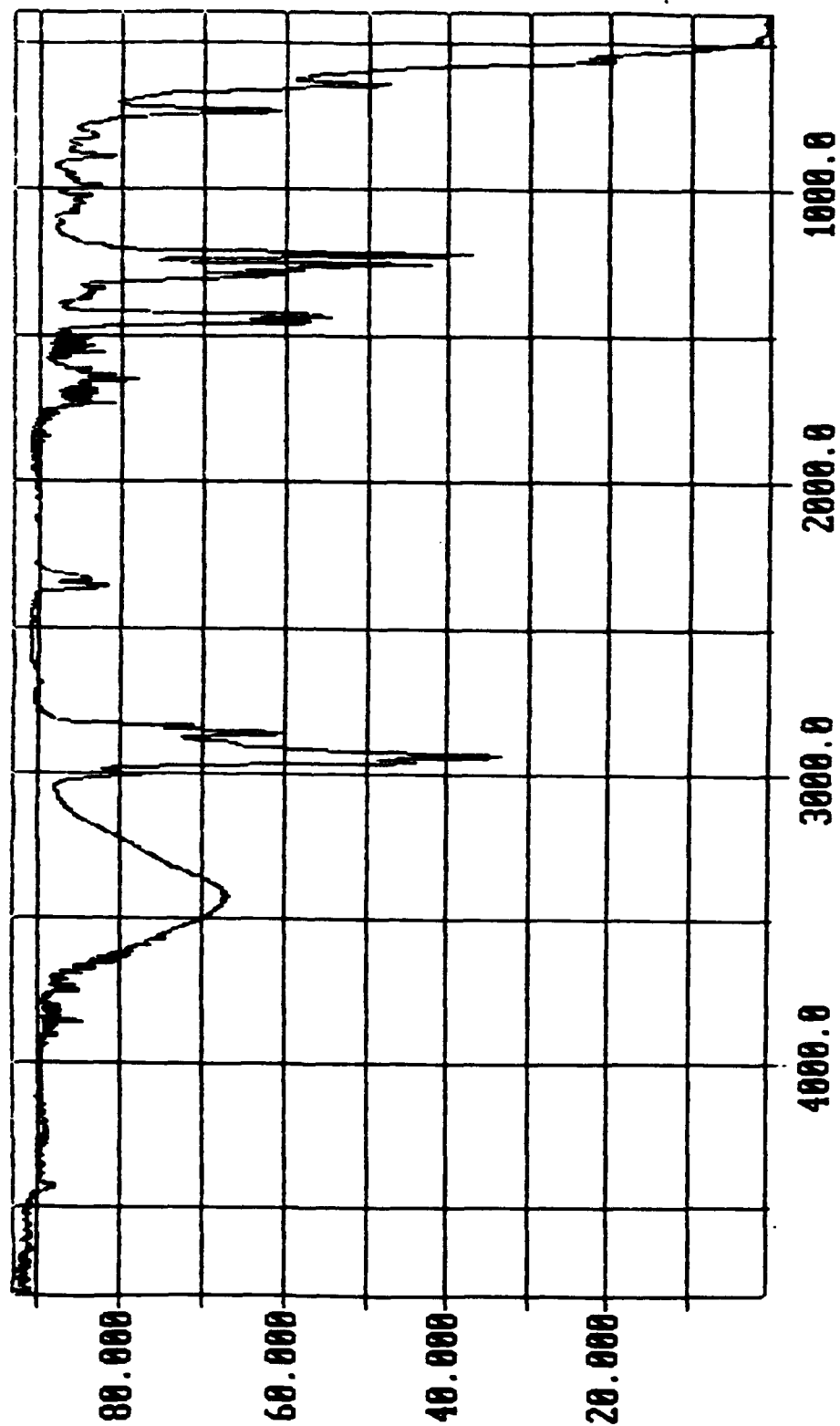
# <sup>13</sup>C NMR [100 MHz] of 1,5-Dibromopentane



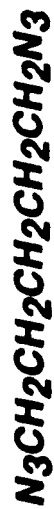
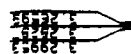
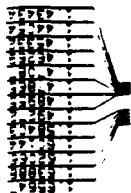
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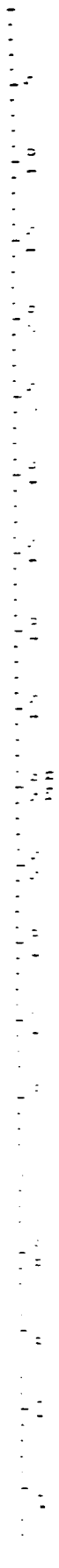
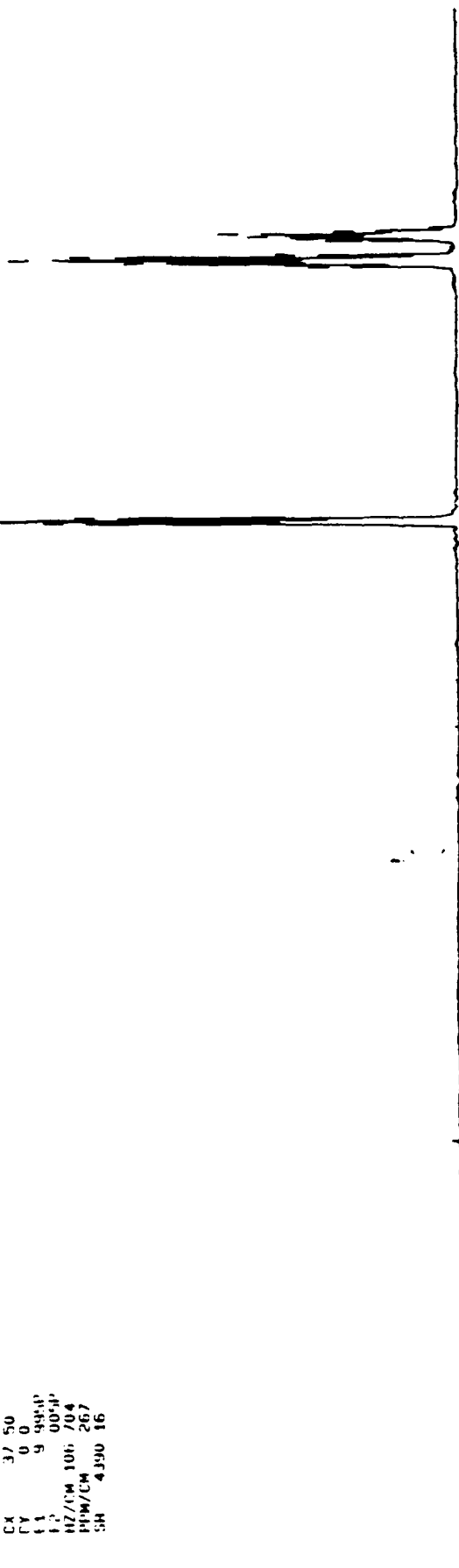
*IR Spectrum of 1,5-Dibromopentane*



**$^1\text{H}$  NMR [400 MHz] of 1,5-Diazidopentane**



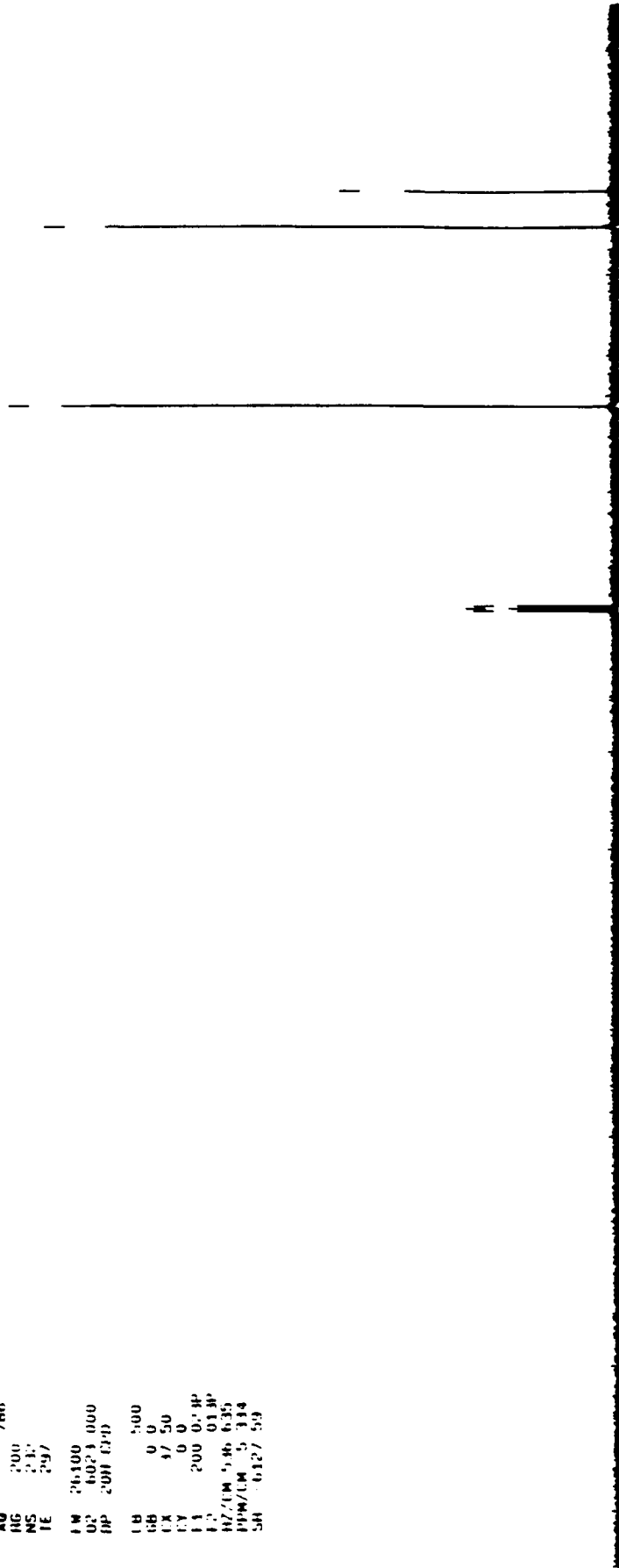
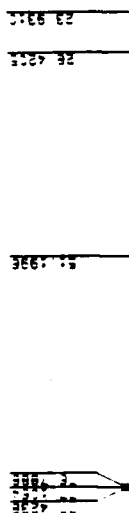
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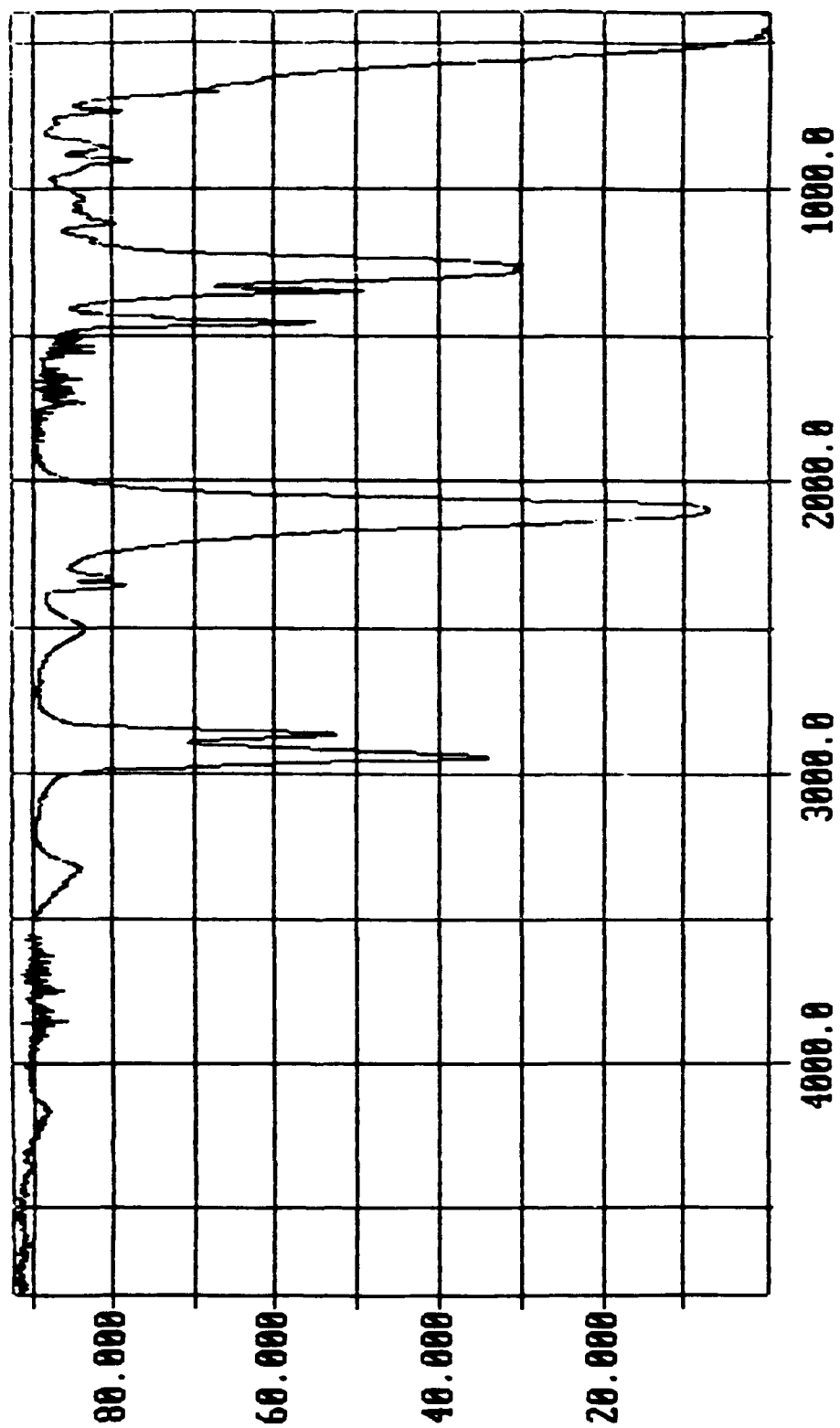
$^{13}\text{C}$  NMR [100 MHz] of 1,5-Diazidopentane



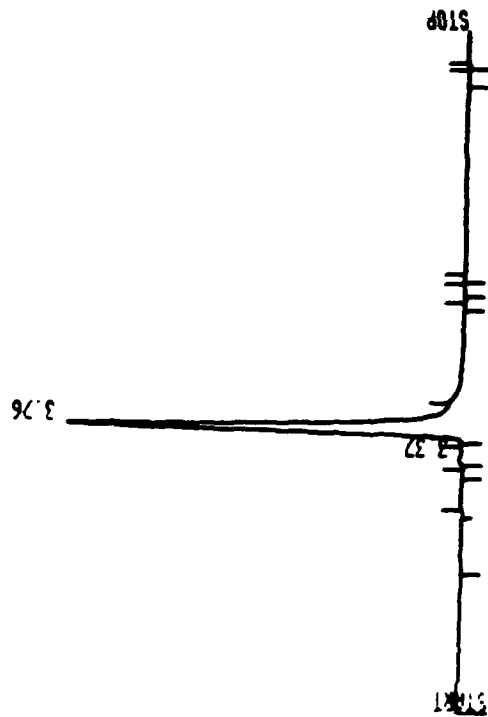
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*IR Spectrum of 1,5-Diazidopentane*



# HPLC of 1,5-Diazidopentane



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WORKFILE NAME:

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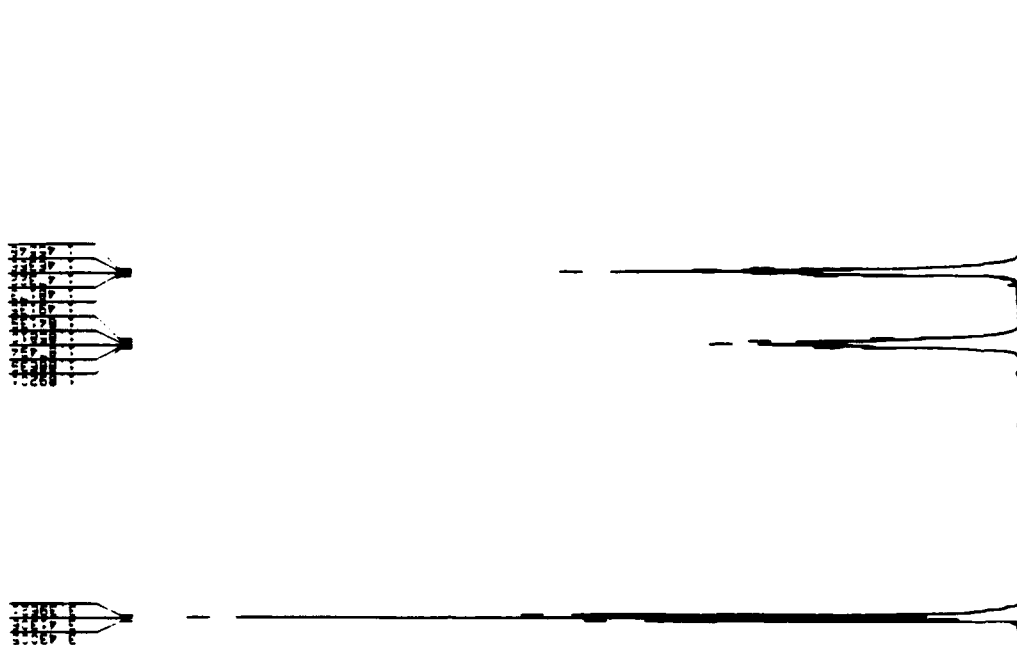
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Column: Zorbax, CN

<sup>1</sup>H NMR [400 MHz] of 1,6-Dibromohexane



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DATE 10 10 89

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31  
1E  
314  
297

100

000	1204	20
001	42	41

(11)  $\text{HNO}_3$  (10)

333

**0000**

100

1990 11 11

0134

6.35  
6.34

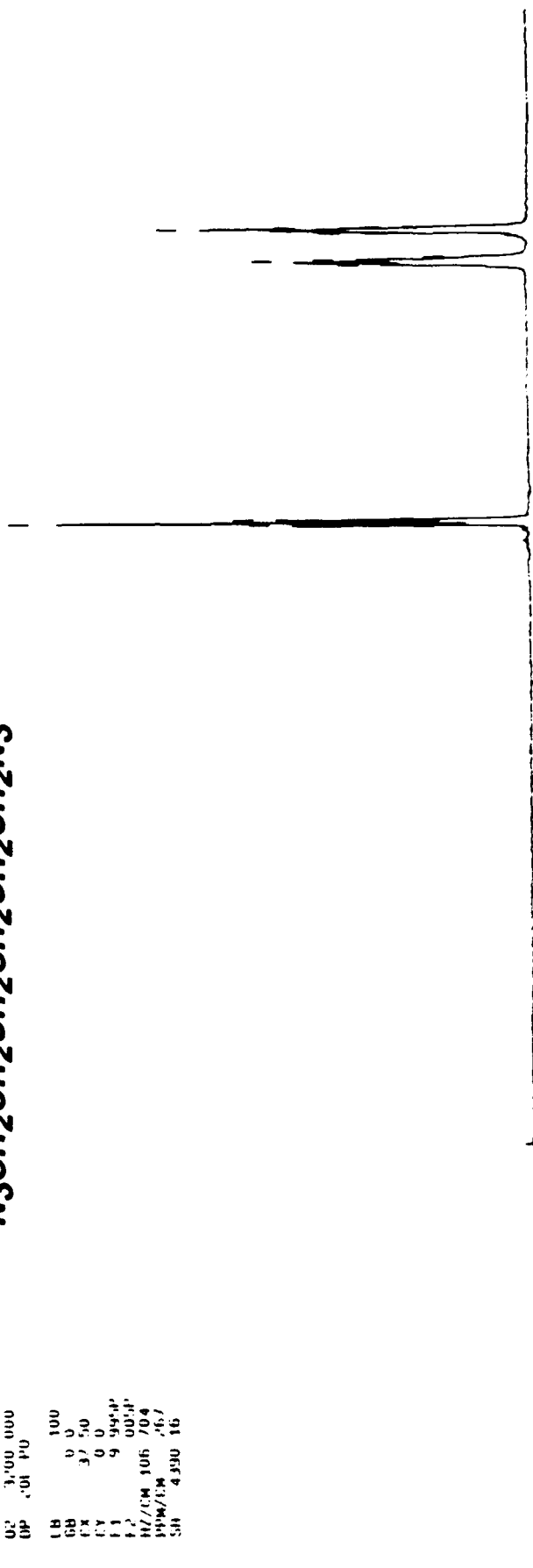
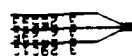
65 / 194 M5

$$\text{Br-CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{Br}$$

$^1\text{H}$  NMR [400 MHz] of 1,6-Diazidohexane



DATE 10 10 89  
 SF 400 134  
 SV 133 0  
 U1 6293 854  
 S1 32/164  
 TD 32/164  
 SM 4203 141  
 WZ/P1 256  
 PM H 0  
 HD 0 0  
 AU 3 899  
 H6 1  
 NS H  
 TE 207  
 FM 5400  
 U2 3100 000  
 DP 204 P0  
 LB 100  
 GB 0 0  
 FX 3750  
 CY 0 0  
 F1 9 9994P  
 F2 0054P  
 HZ/CM 106 704  
 PPM/CM 26.7  
 SN 4390 16



**$^{13}\text{C}$  NMR [100 MHz] of 1,6-Diazidohexane**



DATE 10 10 89

SR 100 h14  
SV 74.0  
Q1 36.8% 148  
S1 32.76H  
T0 3.76H  
SW 20833 133  
HZ/PT 1 272

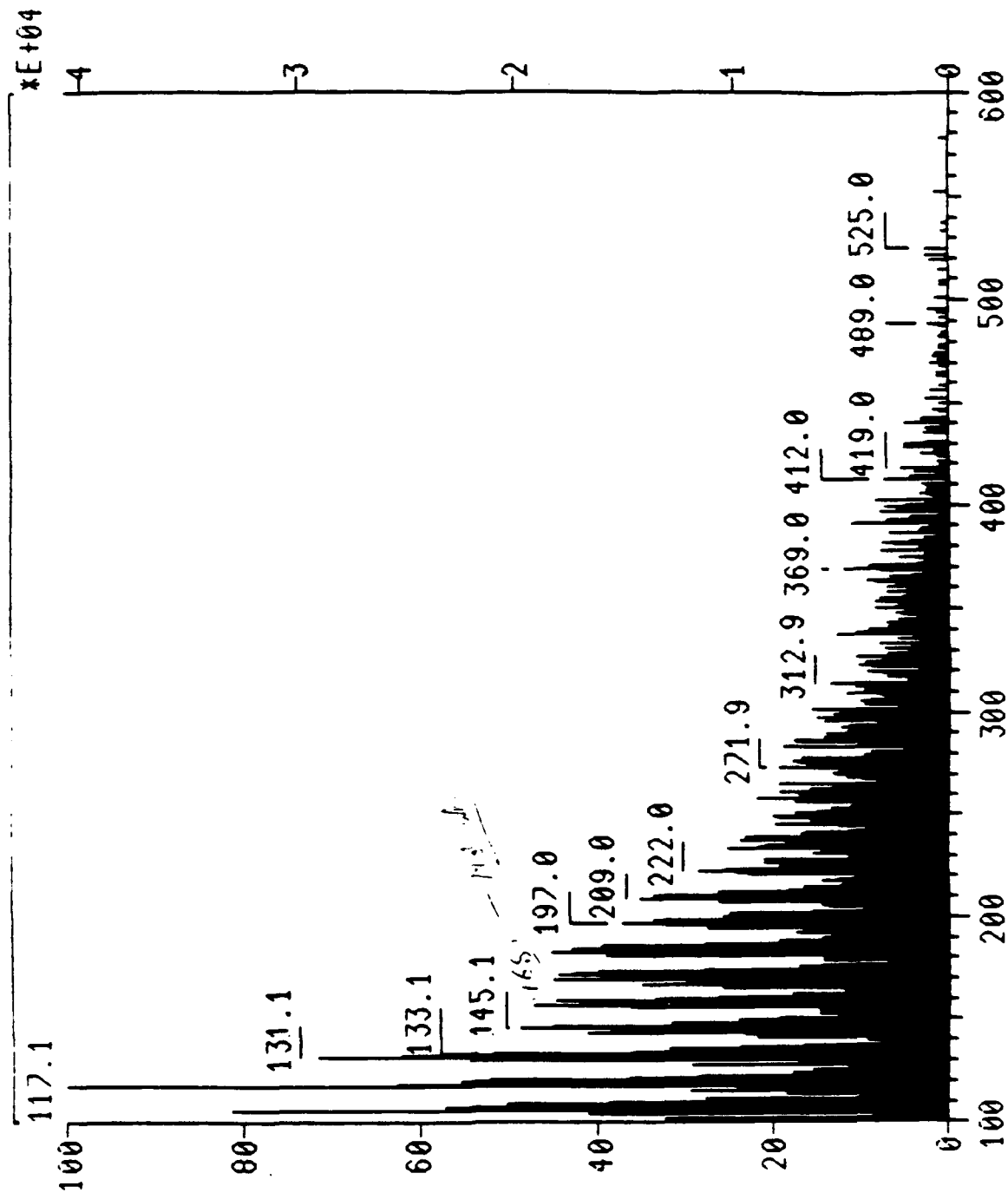
FW 1 4  
HD 0 0  
AU 200 /H6  
RC 200  
TE 297

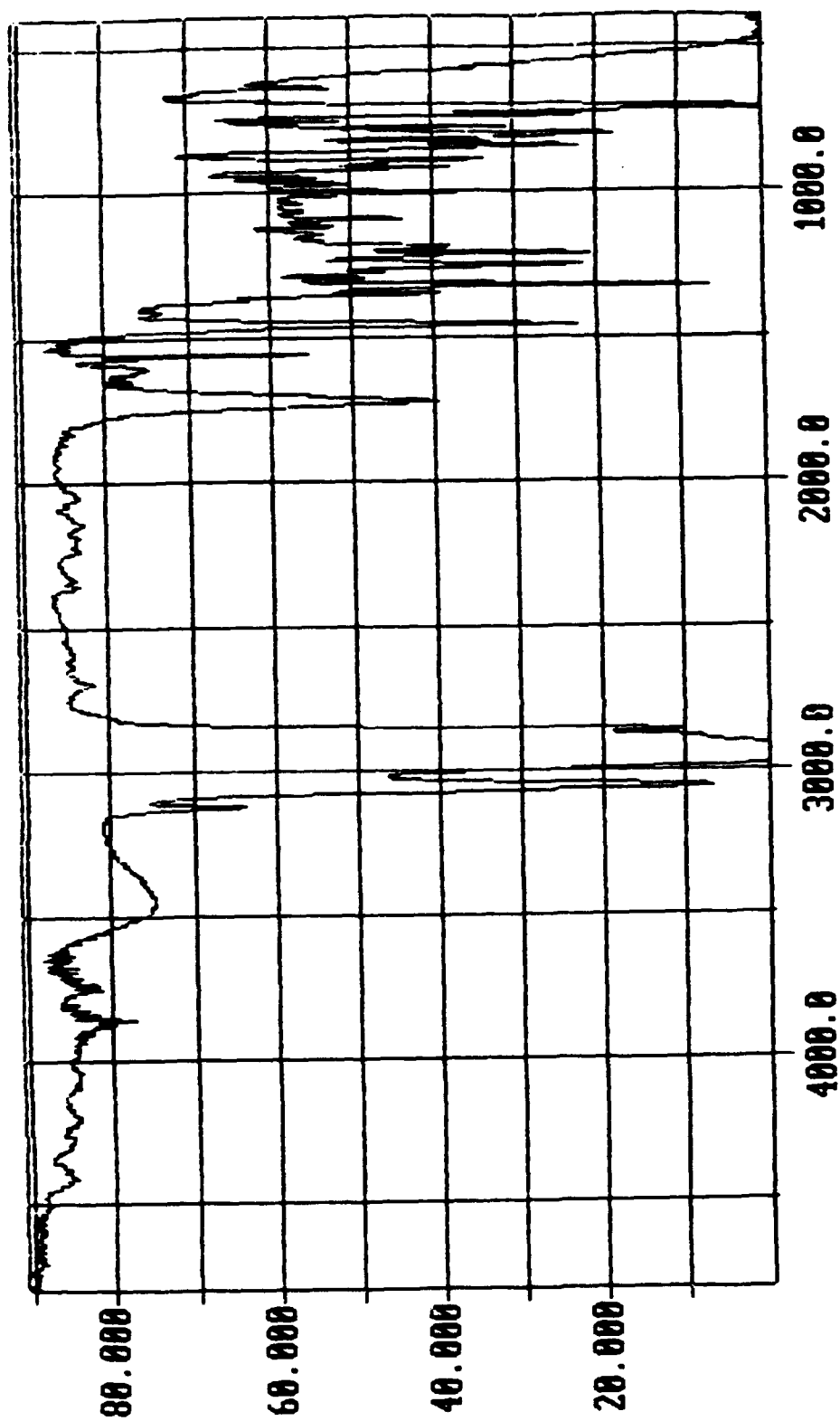
FW 26100  
Q2 6023 000  
DP 20H C10

10 5.00  
60 0 0  
FX 17 50  
13 0 0  
F3 200 0.2 80  
F4 0.1 80  
07/CM 5.86 635  
MM/TM 5 334  
SH 61.27 59



## Mass Spectrum of 1,6-Diazidohexane



*IR Spectrum of Norbornadiene Dimers*

# <sup>1</sup>H NMR [400 MHz] of Norbornadiene Dimers



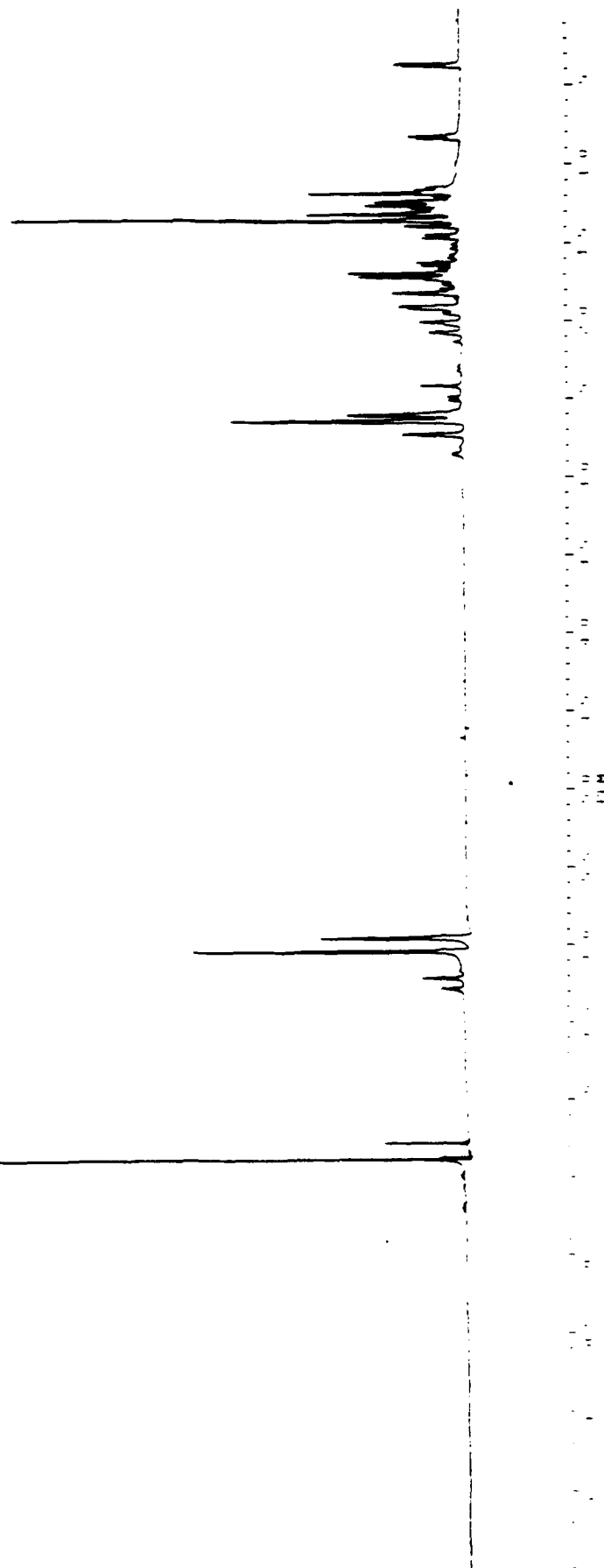
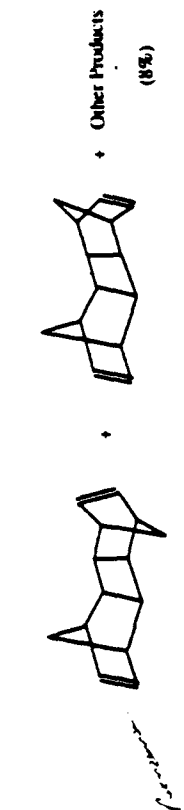
NAME: CHAU, H  
 DATE: 24-4-88

SE: 400 1  
 SY: 131 0  
 U1: 0.511 H  
 SI: 3.768  
 TD: 3.768  
 SW: 4.01 5  
 HZ/PI: 2

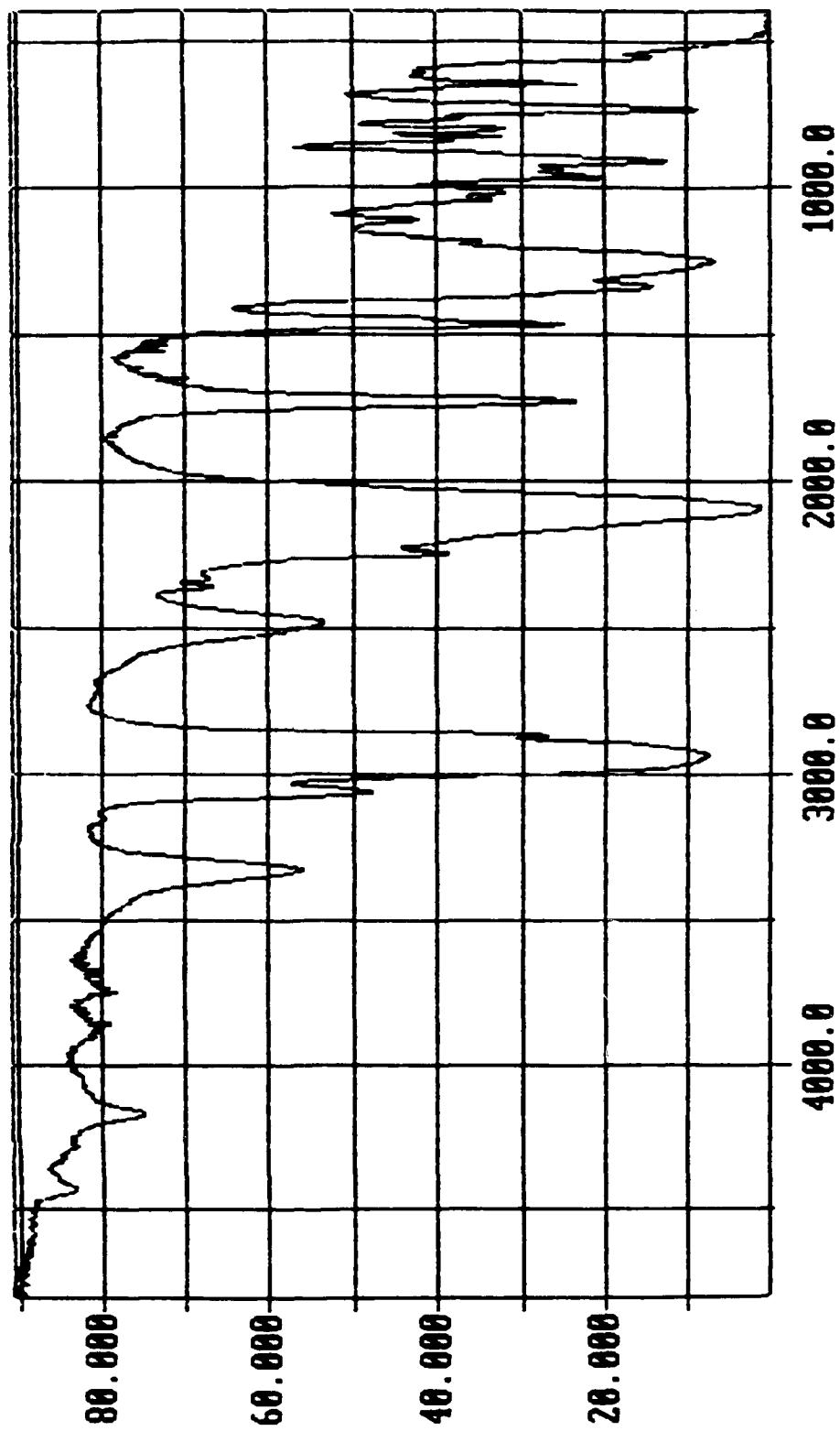
PM: H 0  
 NO: 0 0  
 AD: 3 8  
 HG: 1.7  
 NS: H  
 TE: 297

FM: 5000  
 U2: 3.00 0  
 DP: 201 PD

LB: 1  
 GB: 0 0  
 CX: 37 5  
 CY: 0 0  
 F1: 10 0  
 F2: 0 0  
 HZ/CM: 106.6  
 MPW/CM: 2  
 SN: 4330 1



*IR Spectrum of Tetraazidonorbornane Dimers*



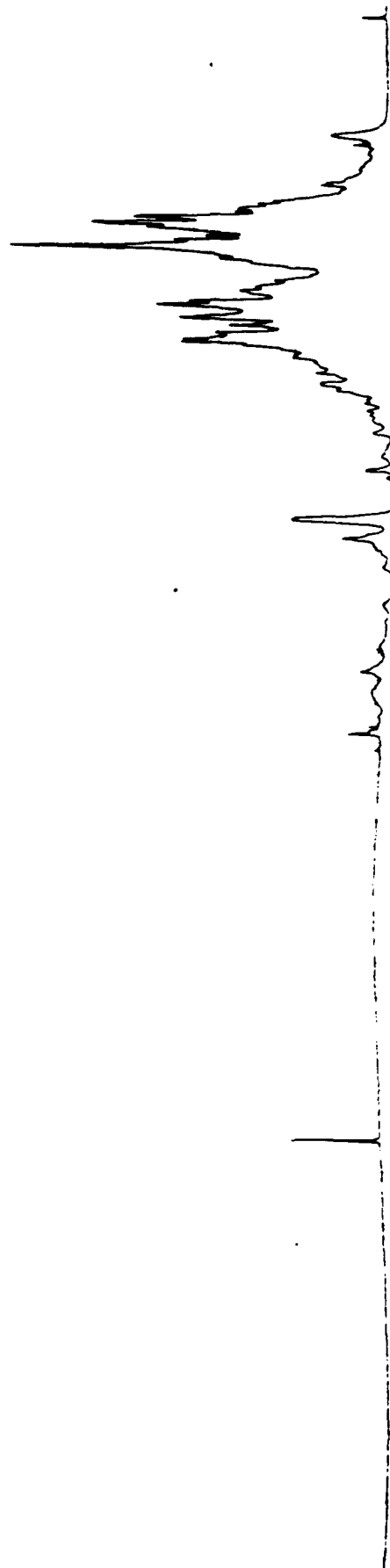
# **<sup>1</sup>H NMR [400 MHz] of Tetraazidonorbornane Dimers**

16



100-100

DATE 10 10  
 SF 400 1  
 SV 133 0  
 O1 6.00 0  
 SI 3.7/0.0  
 TO 3.7/0.0  
 SW 4.01 6  
 HZ/P1 2  
 IN H 0  
 HD 0 0  
 AD 0 0  
 HS 0 0  
 NS H 0  
 TE 0 0  
 FM 5.000  
 U2 3.00 0  
 OP 200 10  
 LB 0 0  
 GB 0 0  
 CX 17 5  
 CY 0 0  
 F1 9 9  
 F2 0 0  
 HZ/LM 100 7  
 HMW/LM 2  
 SH 4390 1

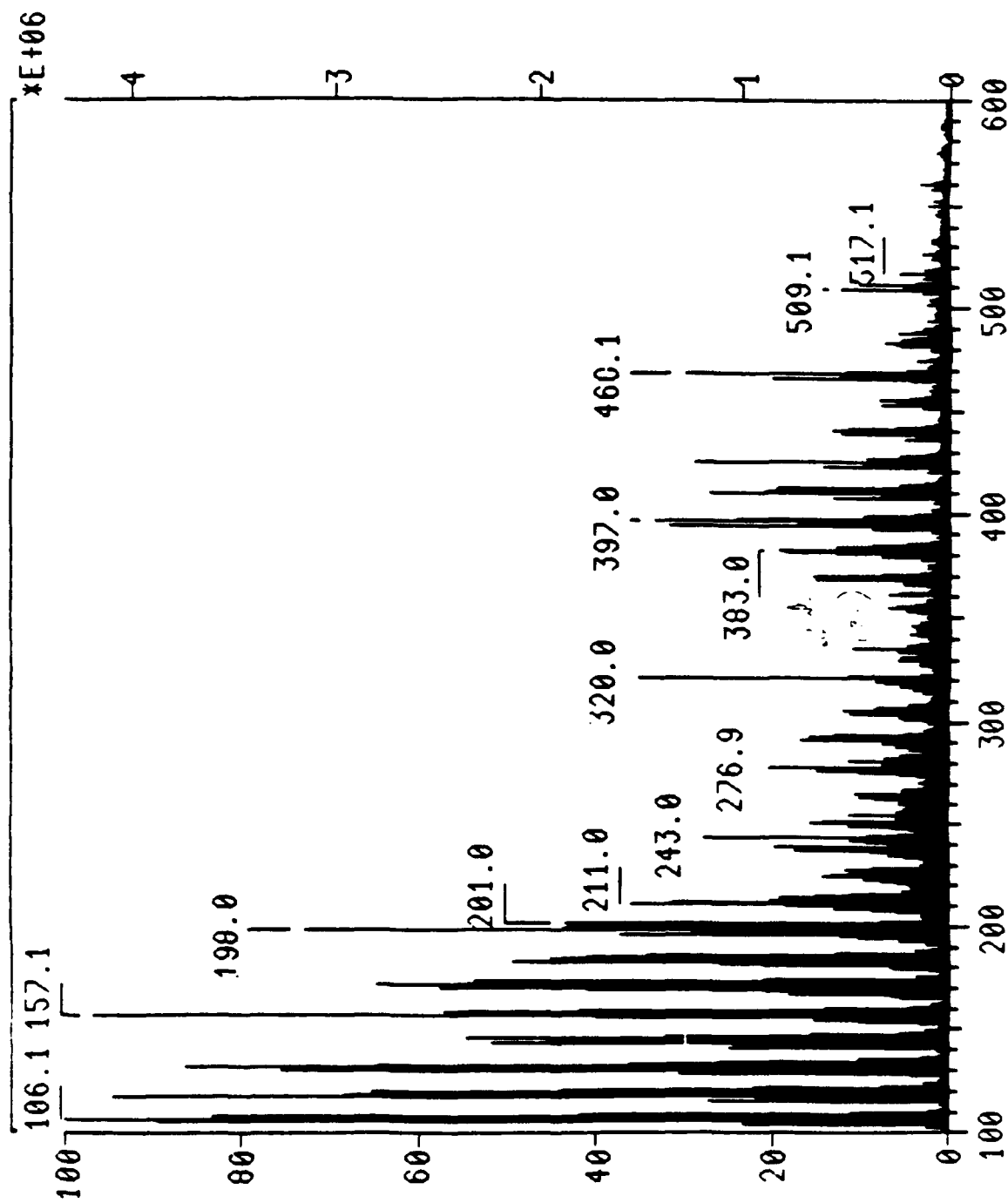


10  
 9  
 8  
 7  
 6  
 5  
 4  
 3  
 2  
 1  
 0



# Mass Spectrum of Tetraazidonorbornane Dimers

17



# <sup>1</sup>H NMR OF DIHYDROBENZVALENE

18

HA0 DIHYDROBENZVALENE

PPM



DATE 25 4 9  
 SF 200 1  
 SV 80 1300  
 OI 3432.0  
 SI 16384  
 TO 16384  
 SW 2702 7  
 HZ/p1 3  
 PW 7 0  
 RD 0 0  
 AQ 3 0  
 RG 16  
 NS 8  
 TE 297  
 FM 3400  
 U2 3284 7  
 DP 63K P0  
 LB 0 0  
 CB 37 5  
 CY 0 0  
 FI 8 0  
 F2 9  
 HZ/CM 48 0  
 PPM/CM 2  
 SH 2342 5

GC OF DIHYDROBENZVALENE

BEST  
AVAILABLE COPY